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SUMMARY

The cryoforming (cryogenic stretch-forming) process for the fabrication of lightweight high-strength stainless-steel pressure vessels was shown in a previous study to be attractive for ambient-temperature service. The purpose of the present investigation was to determine if notch toughness at cryogenic temperatures could be significantly improved by slight compositional modification.

Coupons were cut from a cylindrical pressure vessel cryoformed from a special heat of 301-type stainless steel having low minor element content. Material from the same heat was also obtained which had been stretched in uniaxial (rather than 2 to 1 biaxial) tension. Smooth and sharp-notch tensile specimens made from the coupons were tested at ambient temperature, -320°, and -423° F. Results were compared with previous results for cryoformed AISI 301 and for AISI 301 cold reduced 60 percent. The effect of a stress relief treatment was also determined.

At ambient temperature, the cryoformed special heat showed about the same yield and notch strengths as the previous cryoformed heat. At -320° and -423° F the special heat showed about the same yield strength but considerably higher notch strength than the previous heat. Weld efficiency was slightly above 100 percent at all three test temperatures. The stress relief treatment studied improved both strength and notch toughness at cryogenic temperatures. At 70° and -320° F the strength and notch toughness values for this special heat when cryoformed and stress relieved are superior to those of AISI 301 cold reduced 60 percent ; at -423° F, however, the notch strength of the cryoformed material is somewhat lower.

INTRODUCTION

The cryoforming (cryogenic stretch-forming) process appears attractive for the fabrication of lightweight high-strength stainless-steel pressure vessels, but a previous study (ref. 1) reported poor notch toughness at cryogenic temperatures. In the same study it was suggested that the low-temperature notch toughness of metastable austenitic stainless steels work hardened at subzero temperatures can be improved by a change in the alloy content. The present report evaluates a special 301-type composition having low minor element content for improved low-temperature notch toughness.

The cryoforming process is described in detail in reference 2. A pressure vessel is cryoformed by welding annealed sheet material into an undersize "preform" configuration, then strengthening and sizing by pressurization beyond the elastic limit at -320° F. The mechanical deformation and the metallurgical transformation that occur produce high strength in both the parent metal and the weld, and thus the need for weld reinforcement is eliminated. The finished vessel approaches the monolithic ideal structure of the stress analyst, and the forming operation itself constitutes a type of proof test.

The cryoforming process depends on the ability of metastable austenitic stainless steels to gain strength by transformation to the martensitic phase and the fact that this transformation is more easily accomplished by deformation at low temperature. The amount of transformation that occurs is an interrelated function of chemical composition, temperature, and strain level, as discussed in references 3 and 4. However, the effects of these variables on low temperature toughness are not fully understood at present.

Reference 1 presented the results of a program evaluating the strength and toughness of cryoformed AISI 301 stainless steel at temperatures from ambient to -423° F. Tensile strength and weld efficiency were found to be high at all test temperatures, but poor notch toughness was observed at -423° F.

Research by other investigators (refs. 5 and 6) indicated that the low-temperature toughness of metastable austenitic stainless steels may be strongly influenced by their alloying elements. For example, variations that are within the range of the AISI specification for 301 can significantly alter the low-temperature notch strength and fracture toughness. In these studies mechanical work was introduced by subzero rolling rather than stretching. Only a few tests were made at -320° F, and none below that temperature. However it appeared that high carbon, manganese, and silicon contents were detrimental to low-temperature toughness.

The purpose of the present investigation was to determine if the poor low-temperature toughness of cryoformed 301 stainless steel reported in reference 1 can be significantly improved by changing the composition. A cylindrical pressure vessel was obtained which had been cryoformed from a special heat of 301-type stainless steel having low minor element content. The vessel was cut into coupons which were machined to smooth and sharp-notched tensile specimen configurations and tested at ambient, liquid nitrogen, and liquid hydrogen temperatures. Results are compared with those of reference 1 and also with those for material from the same heat which was stretched in uniaxial (rather than

2 to 1 biaxial) tension. The effect of a stress relief treatment on tensile properties was also determined.

MATERIALS AND TEST SPECIMENS

Biaxially cryoformed material was received in the form of a cylindrical tank with hemispherical ends. The tank had been pressurized at -320° F to a true hoop stress of 277 ksi, which resulted in a permanent diametral forming strain of 7.1 percent. Dimensions and forming parameters are given in table I. Tensile coupons oriented in both the hoop and the axial directions were cut from the cylindrical portion of the tank, as illustrated in figure 1. The coupons were then machined to the configurations shown in figure 2. No attempt was made to flatten the specimens or to smooth the weld bead.

TABLE I. - VESSEL PARAMETERS

Before forming	
Outside diameter, in.	11.025
Wall thickness, in.	0.0376
Cylinder length, in.	33
After forming	
Outside diameter, in.	11.810
Forming strain, percent	7.1
Wall thickness, in.	0.0361
Forming pressure, psig	1650
Forming stress (hoop), ksi	277

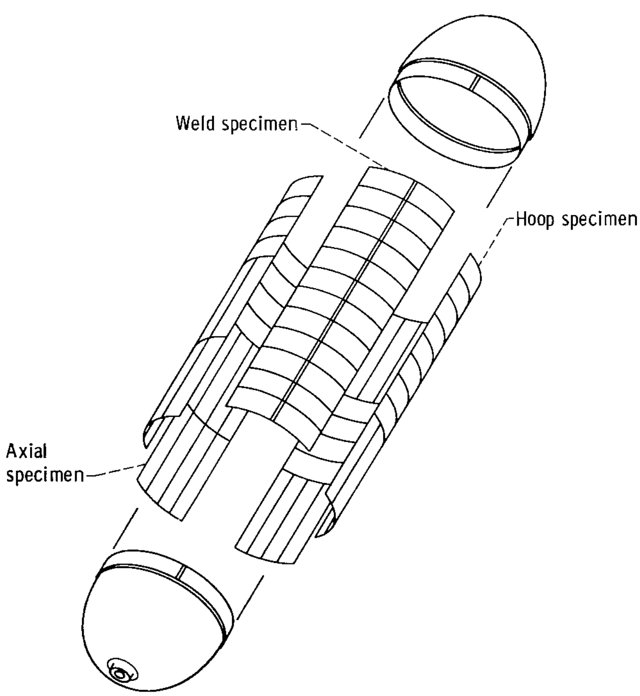


Figure 1. - Cutting of tensile coupons from cylindrical tank.

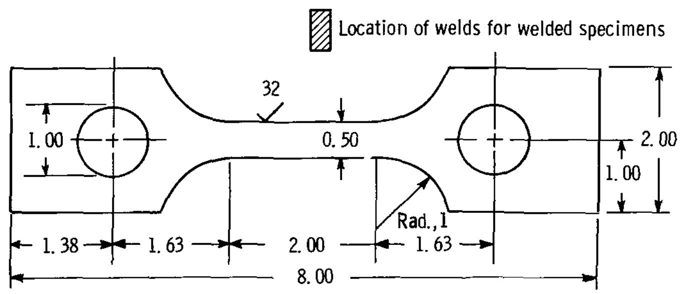
The uniaxially stretched material from the same heat when received resembled oversized smooth tensile specimens and had been stretched at -320° F to a true stress of 240 ksi (forming strain, 11.3 percent). These were then machined to size and notched as shown in figure 2. For all notched specimens the notch radii were not greater than 0.0007 inch (theoretical elastic stress-concentration factor $K_t = 21$). Following machining, specimens to be stress relieved were held at 790° F for 20 hours.

Chemical compositions of the alloys studied and referenced are presented in table II along with the limits of the AISI specification of 301. Heat 40226 is the subject of this report; heat 58044 is the cryoformed 301 studied in reference 1; heat 97838 (also from ref. 1) is 301 conventially cold-reduced 60 percent at ambient temperature. Heat 40226 contains

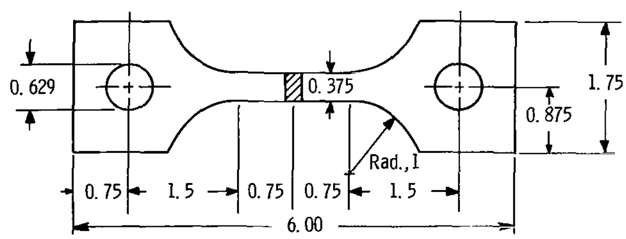
TABLE II. - COMPOSITION OF HEATS TESTED AND REFERENCED

[Analysis made on finished sheet]

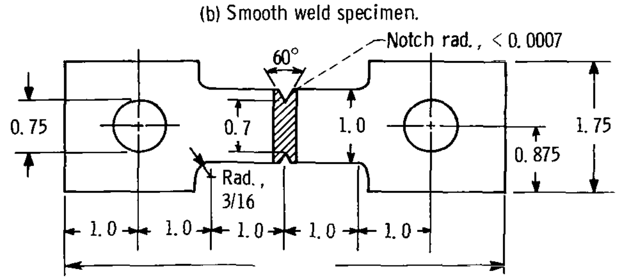
Heat	Sulfur	Phosphorus	Carbon	Manganese	Silicon	Chromium	Nickel	Nitrogen	Oxygen
	Percent by weight								
40226 (Present study)	0.006	0.006	0.032	0.04	0.01	18.90	7.65	0.015	0.010
58044 (ref. 1; cryoformed)	.015	.027	.075	1.55	.41	17.19	7.53	.045	.018
97838 (ref. 1; cold reduced)	.013	.024	.11	1.02	.38	17.43	7.21	.044	.022
AISI specification for 301	.030 (maximum)	.045 (maximum)	.15 (maxinum)	2.00 (maximum)	1.00 (maximum)	16.0 to 18.0	6.0 to 8.0	Not specified	Not specified



(a) Smooth parent metal specimen.



(b) Smooth weld specimen.



(c) Notched weld or parent metal specimen.

Figure 2. - Smooth and sharp-notch sheet tensile specimens. (All dimensions in inches unless otherwise specified.)

slightly more chromium than the 18 percent maximum, although in all other elements it is within the AISI specification for 301. The minor element contents are extremely low and are considerably less than what might be considered typical.

APPARATUS AND PROCEDURE

Specimens were tested in a universal testing machine. Strain was measured by using a clamp-on differential-transformer extensometer of 2-inch gage length and an autographic stress-strain recorder. The extensometer was previously calibrated at all three test temperatures with a micrometer-driven calibration device.

Cryogenic test temperatures were established by immersing the specimen in liquid nitrogen or liquid hydrogen. A vacuum-jacketed cryostat was used to minimize boiloff. Correct cryogenic temperature was assured by maintaining the liquid level several inches above the upper specimen grip. Liquid level sensing was accomplished by means of a carbon resistor.

The smooth tensile strength, the yield strength (0.2 percent offset), and the notch tensile strength were determined at ambient temperature, -320°, and -423° F in the as-received and stress-relieved conditions. Weld ultimate strength was determined only for material in the as-received condition.

RESULTS AND DISCUSSION

The average tensile properties for the cryoformed special heat 40226 appear in table III (p. 11) along with comparable data from reference 1 for the normal-composition heat 58044. In most cases these represent the average of three specimens tested per condition. The complete data obtained in this investigation are listed in table IV (pp. 12 to 14).

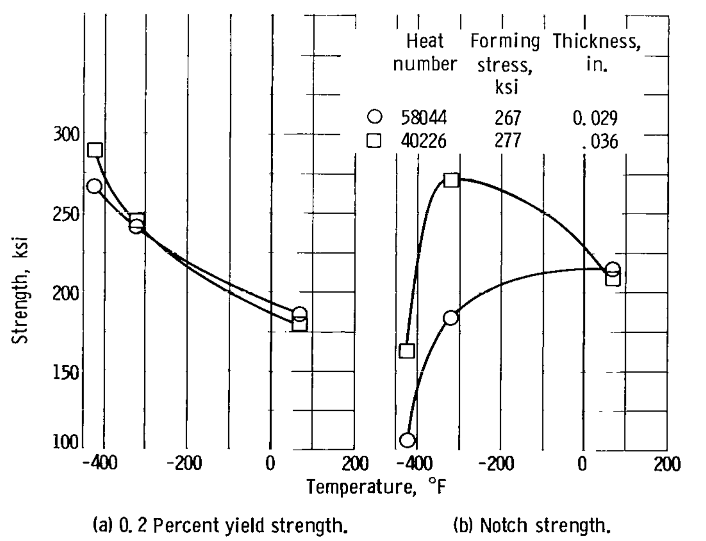


Figure 3. - Comparison of special heat (40226) with previous heat (58044). (Specimens from hoop direction; not stress relieved.)

Comparison of Special Heat and Previous Heat

In figure 3 the yield and notch strengths (cryoformed) of the two heats are compared. The difference between yield strengths is small, with special heat 40226 being about 25 ksi stronger at -423° F than heat 58044. The difference between room-temperature notch strengths is also small. There is however a considerable difference between their notch strengths at cryogenic temperatures. The notch strength of special heat 40226 is about 90 ksi greater at -320° F and 55 ksi greater at -423° F than the previous heat 58044, which had a more normal composition.

The tanks from which the coupons were cut were as nearly identical as possible, as were the specimen geometries and manner of testing. The only significant difference between heats is composition. Thus it is reasonable to assume that the difference between notch strengths at cryogenic temperatures is due to the difference in composition between the two heats. The composition of this special heat may not necessarily represent the ideal composition for cryoforming and further improvement in low-temperature properties might result from further development.

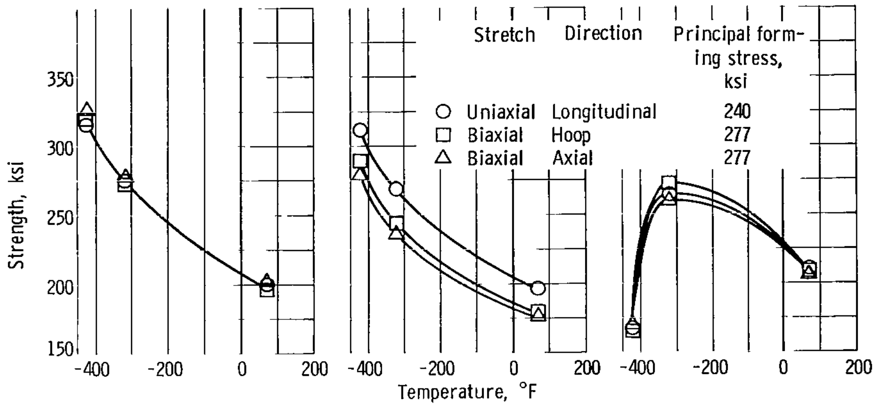
Comparison of Uniaxial and Biaxial Stretch

For the purposes of alloy development and screening it would be both cumbersome and expensive to cryoform a separate pressure vessel for each heat and forming stress level to be investigated. It would be desirable if the properties of material stretched in uniaxial tension in a conventional tensile machine and cryostat could be related to those of material stretched in biaxial tension. A theoretical correlation does not appear feasible, and furthermore one would not expect that the differing plastic flows which occur in different stress fields would result in identical material properties after stretching.

According to the supplier, however, similar room-temperature ultimate strengths are obtained if a material is stretched in different stress fields to the same effective true stress. To compare ultimate strengths at cryogenic temperatures and notch strengths at all three temperatures for this heat, additional material from the same heat was obtained which had been stretched by the supplier in uniaxial tension at -320° F to 240 ksi true stress (The effective stress for a cylinder is $\frac{1}{2}\sqrt{3}$ times the hoop stress, thus $277 \times 0.866 = 240.$). The properties of this material are compared in figure 4 with those of material cut from the pressure vessel in both the hoop and the axial directions.

The ultimate and notch strengths are seen to agree very well over the entire temperature range. The yield strength curve for the uniaxially stretched material has the same shape as that of the biaxially stretched material from the same heat but is about 20 ksi higher over the entire range. During the initial stretch forming at -320° F, the uniaxially stretched material underwent more elongation than the biaxially stretched material (11.3 as opposed to 7.1 percent); thus a greater amount of martensite should have formed and higher yield strength upon subsequent testing might be expected.

Although good correlation is seen here, additional data would be required to deter-



(a) Ultimate strength. (b) Yield strength. (c) Notch strength.

Figure 4. - Comparison of properties obtained from uniaxially stretched specimens and specimens cut from tank. (Heat 40226; thickness, 0.036 inch; not stress relieved.)

mine if equally good results can be obtained for other heats of material and other forming stress levels. As the forming strains reported here were rather low (about 7 and 11 percent for the biaxially and uniaxially stretched materials, respectively), it would be reasonable to expect greater differences between uniaxial and biaxial stretch at higher forming strain levels.

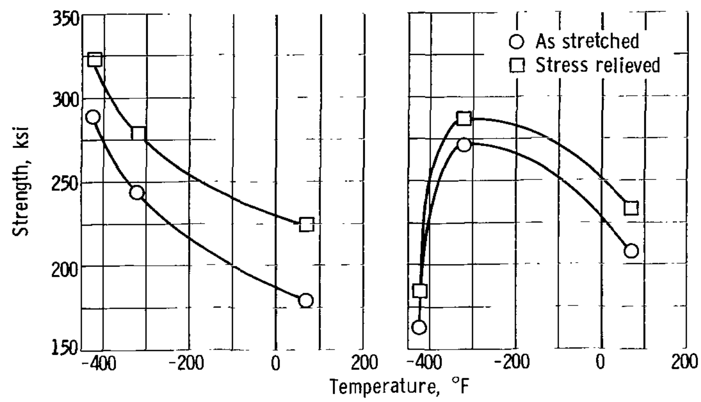
Effect of a Stress Relief Treatment

Thermal treatments on the order of 24 hours at 800° F are often used after cold reduction or cold forming of 300-series stainless steels, and are usually referred to as stress-relief treatments. In addition to relaxation of residual stresses, however, additional strengthening sometimes occurs which is probably due to a form of precipitation hardening. Reference 5 contains some interesting observations and discussion on this subject. At temperatures much in excess of 800° F, however, a reduction in strength may occur, as was found in reference 7.

The effect of 20 hours at 790° F on the yield and notch strengths of the special heat can be seen in figure 5. The treatment increases the notch strength and the yield strength at all three temperatures. For this composition and forming stress level, such a treatment would be beneficial.

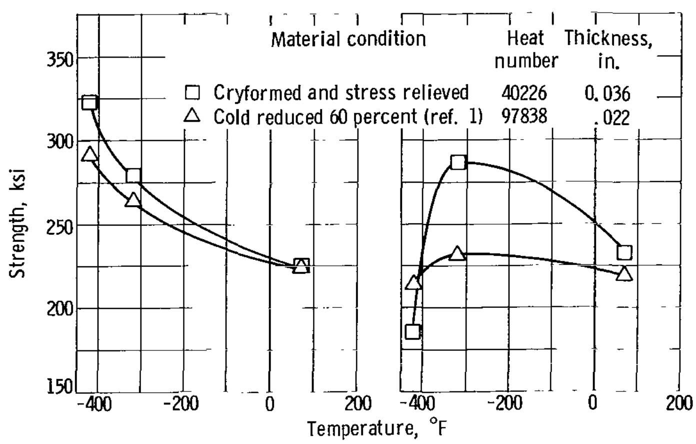
Comparison with Cold-Reduced AISI 301

In figure 6 the special heat when cryoformed and stress relieved is compared with AISI 301 conventionally cold reduced 60 percent (ref. 1). At room temperature the cryo-



(a) 0.2 Percent yield strength. (b) Notch strength.

Figure 5. - Effect of stress relief on cryoformed stainless steel. (Heat 40226; hoop direction.)



(a) 0.2 Percent yield strength. (b) Notch strength.

Figure 6. - Comparison of cryoformed 301-type and cold reduced 301 stainless steels. (In direction of maximum mechanical work.)

formed material has about the same yield strength as and slightly higher notch strength than the cold-reduced material; at -320° F, slightly higher yield strength and considerably higher notch strength; and at -423° F, higher yield strength and lower notch strength.

In evaluating materials for a specific application one must consider not only the basic properties of the materials but also the extent to which the materials are modified during fabrication processes. The data presented for the cryoformed material represent the "as-fabricated" condition whereas the data for the cold-reduced material represent only the basic "pre-fabrication" condition. For pressure vessel applications, therefore, the cryoformed material shows considerable promise.

Weld Efficiency

Examination of table III (p. 11) reveals that weld-to-parent ultimate tensile strength ratio or "weld efficiency" was slightly greater than unity at all three test temperatures for the as-received condition. These values are somewhat higher than those obtained for the previous heat 58044, but this difference is not felt to be significant as all failures were well away from the weld zone. Lack of sufficient material prevented determination of weld efficiency for the stress-relieved condition.

Weld-to-parent notch strength ratios were also above unity for all test conditions except for the stress-relieved specimens at -320° F. Whether this lower value was due to increased notch sensitivity or to experimental scatter could not be determined for lack of sufficient material. These weld data represent the averages of only two specimens per test condition and should be considered accordingly.

SUMMARY OF RESULTS

The results of this investigation indicate that the special 301-type stainless steel having low minor element content is superior to the heat previously studied (NASA TN D-2202) for the cryogenic stretch forming of pressure vessels for service at cryogenic temperatures. Results may be summarized as follows:

1. The special heat 40226 when cryoformed showed approximately the same yield strength as the previous heat 58044 from ambient temperature to -423° F.

2. The special heat 40226 when cryoformed showed about 48 percent greater sharp-notch strength at -320° F and 54 percent greater at -423° F than the previous heat 58044.

3. Weld efficiency was excellent, being slightly above 100 percent at all three test temperatures.

4. At 70° and -320° F strength and notch toughness values for this heat when cryoformed and stress relieved are superior to those of AISI 301 cold reduced 60 percent. At -423° F, however, the notch strength of the cryoformed material is somewhat lower.

5. The standard stress relief treatment proved beneficial to both strength and notch toughness at cryogenic temperatures.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 10, 1966.

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TABLE III. - AVERAGE TENSILE PROPERTIES OF TWO HEATS OF CRYOFORMED STAINLESS STEEL (HEAT 58044 DATA FROM REF. 1)

Heat number	Material thickness, in.	Type of stretch	Forming stress, ksi	Forming strain, percent	Thermal treatment	Specimen orientation	Test temperature, $^{\circ}F$	Parent metal							Welds			
								Ultimate tensile strength, ksi	0.2 Percent yield strength, ksi	Notch tensile strength, ksi	Elastic modulus, psi	Elongation in 2 inches, percent	Notch-to-yield strength ratio	Nominal fracture toughness, ksi $\sqrt{in.}$	Weld ultimate strength, ksi	Weld notch strength, ksi	Weld-to-parent ultimate strength ratio	Weld-to-parent notch tensile strength ratio
58044	0.029	Biaxial	267	14.8	None	Hoop	70	222	186	215	24.9×10^{6}	(a)	1.156	(b)	221	216	0.995	1.005
							-320	304	241	184	29.5	(a)	.764	111	303	169	.997	.918
							-423	332	267	106	26.2	(a)	.397	59	316	97	.952	.915
						Axial	70	224	168	184	27.8×10^{6}	(a)	1.094	(b)	(a)	(a)	(a)	(a)
							-320	304	213	105	29.5	(a)	.491	59	(a)	(a)	(a)	(a)
							-423	310	232	59	29.1	(a)	.255	32	(a)	(a)	(a)	(a)
40226	0.036	Biaxial	277	7.1	None	Hoop	70	196	180	208	24.3×10^{6}	6	1.159	(b)	201	225	1.026	1.082
							-320	272	244	272	27.9	$10\frac{1}{2}$	1.115	(b)	277	275	1.018	1.011
							-423	320	289	163	26.7	$10\frac{1}{2}$	.564	93	330	(a)	1.031	(a)
						Axial	70	202	176	206	26.4×10^{6}	5	1.170	(b)	(a)	(a)	(a)	(a)
							-320	278	237	259	28.0	9	1.096	(b)	(a)	(a)	(a)	(a)
							-423	326	279	168	27.3	$9\frac{1}{2}$	.603	97	(a)	(a)	(a)	(a)
					790° F for 20 hr	Hoop	70	231	225	233	25.3×10^{6}	4	1.036	(b)	(a)	258	(a)	1.252
							-320	291	279	287	27.4	9	1.028	(b)	(a)	252	(a)	.878
							-423	335	323	185	28.8	2	.572	106	(a)	185	(a)	1.000
						Axial	70	235	221	232	27.7×10^{6}	4	1.050	(b)	(a)	(a)	(a)	(a)
							-320	297	285	264	28.6	$6\frac{1}{2}$	.925	171	(a)	(a)	(a)	(a)
							-423	345	328	189	30.4	$4\frac{1}{2}$	.576	108	(a)	(a)	(a)	(a)
	0.037	Uniaxial	240	11.3	None	Longitudinal	70	200	197	210	25.4×10^{6}	5	1.066	(b)	(a)	(a)	(a)	(a)
							-320	275	269	264	25.5	7	.981	187	(a)	(a)	(a)	(a)
							-423	317	312	167	26.8	3	.535	99	(a)	(a)	(a)	(a)

[a]No data taken.

[b]Ductile fracture; fracture toughness criterion does not apply.

TABLE IV. - EXPERIMENTAL DATA FOR HEAT 40226 CRYOFORMED

Type of stretch	Thermal treatment	Orientation	Test temperature, °F	Ultimate tensile strength, ksi	0.2 Percent yield strength, ksi	Elastic modulus, psi	Elongation, percent	Notch strength, ksi
Biaxial	None	Hoop	70	196.3	178.0	24.9×10^6	6	211.2
				199.1	181.9	24.4	6	205.4
				193.6	179.4	23.8	6	(a)
				[b]196.3	[b]179.8	[b]24.4×10^6	[b]6	[b]208.3
			-320	271.2	244.6	27.2×10^6	11	258.6
				270.6	241.5	27.7	10	274.7
				273.4	246.6	28.8	$10\frac{1}{2}$	283.5
				[b]271.7	[b]244.2	[b]27.9×10^6	[b]$10\frac{1}{2}$	[b]272.3
			-423	320.1	293.5	27.2×10^6	10	173.9
				319.0	282.9	26.4	$11\frac{1}{2}$	163.4
				319.4	291.5	26.9	$10\frac{1}{2}$	152.5
				[b]319.5	[b]289.3	[b]26.8×10^6	[b]$10\frac{1}{2}$	[b]163.3
		Axial	70	201.6	177.1	26.7×10^6	5	207.5
				201.9	174.5	26.0	5	205.2
				(c)	(c)	(c)	(c)	203.8
				[b]201.8	[b]175.8	[b]26.4×10^6	[b]5	[b]205.5
			-320	276.6	233.7	28.4×10^6	$8\frac{1}{2}$	266.3
				278.6	242.8	26.9	9	249.7
				277.9	233.2	28.8	9	261.7
				[b]277.7	[b]236.6	28.0×10^6	[b]9	[b]259.2
			-423	323.9	274.9	26.8×10^6	$9\frac{1}{2}$	168.1
				328.2	282.5	27.7	$9\frac{1}{2}$	173.4
				(d)	(d)	(d)	(d)	162.6
				[b]326.1	[b]278.7	[b]27.3×10^6	[b]$9\frac{1}{2}$	[b]168.0

[a]Failed in head.
[b]Average value.
[c]No data taken.
[d]Data rejected because of excessive deviation from mean.

TABLE IV. - Continued. EXPERIMENTAL DATA FOR HEAT 40226 CRYOFORMED

Type of stretch	Thermal treatment	Orientation	Test temperature, °F	Ultimate tensile strength, ksi	0.2 Percent yield strength, ksi	Elastic modulus, psi	Elongation, percent	Notch strength, ksi
Biaxial	790° F for 20 hr	Hoop	70	231.2	225.7	25.4×10^6	4	233.8
				232.3	225.7	25.2	$3\frac{1}{2}$	234.1
				230.4	223.8	25.4	4	232.0
				[b]231.3	[b]225.1	[b]25.3×10^6	[b]4	[b]233.3
			-320	284.8	270.4	27.3×10^6	$14\frac{1}{2}$	287.8
				293.4	283.6	27.4	$7\frac{1}{2}$	291.6
				294.0	283.6	27.6	6	281.5
				[b]290.7	[b]279.2	[b]27.4×10^6	[b]9	[b]287.0
			-423	333.3	322.4	28.9×10^6	2	184.3
				333.0	323.8	28.8	2	178.3
				337.3	323.7	28.8	$1\frac{1}{2}$	191.7
				[b]334.5	[b]323.3	[b]28.8×10^6	[b]2	[b]184.8
		Axial	70	234.1	220.7	28.0×10^6	$4\frac{1}{2}$	237.6
				234.8	(e)	27.3	4	234.8
				(c)	(c)	(c)	(c)	223.0
				[b]234.5	[b]220.7	[b]27.7×10^6	[b]4	[b]231.8
			-320	298.3	286.0	29.5×10^6	$6\frac{1}{2}$	272.5
				295.7	284.0	27.5	6	258.3
				298.2	285.9	28.8	$5\frac{1}{2}$	261.3
				[b]297.4	[b]285.3	[b]28.6×10^6	[b]6	[b]264.0
			-423	345.9	323.2	31.6×10^6	5	195.0
				344.0	330.5	30.9	$4\frac{1}{2}$	201.4
				345.7	329.4	28.8	$1\frac{1}{2}$	169.9
				[b]345.2	[b]327.7	[b]30.4×10^6	$4\frac{1}{2}$	[b]188.8

[b]Average value.
[c]No data taken.
[e]Faulty extensometer trace.

TABLE IV. - Concluded. EXPERIMENTAL DATA FOR HEAT 40226 CRYOFORMED

Type of stretch	Thermal treatment	Orientation	Test temperature, °F	Ultimate tensile strength, ksi	0.2 Percent yield strength, ksi	Elastic modulus, psi	Elongation, percent	Notch strength, ksi
Uniaxial	None	Longitudinal	70	200.8	197.8	25.0×10^6	4	208.1
				201.1	198.3	25.9	5	209.1
				197.8	194.5	24.9	6	213.8
				[b]199.9	[b]196.9	[b]25.4×10^6	[b]5	[b]210.3
			-320	274.9	268.2	26.9×10^6	8	257.8
				272.6	266.2	26.6	7	268.3
				276.3	271.5	25.9	6	266.2
				[b]274.6	[b]268.6	[b]25.5×10^6	[b]7	[b]264.1
			-423	312.8	312.0	26.8×10^6	1	159.8
				324.2	313.4	26.9	7	169.9
				313.9	311.1	26.7	1	170.5
				[b]317.0	[b]312.2	[b]26.8×10^6	[b]3	[b]166.7
Biaxial	None	Hoop weld	70	201.4	(c)	(c)	3	225.5
				199.9	(c)	(c)	$2\frac{1}{2}$	223.9
				[b]200.7	---	---	[b]$2\frac{1}{2}$	[b]224.7
			-320	278.2	(c)	(c)	2	275.2
				276.6	(c)	(c)	2	274.2
				[b]277.4	---	---	[b]2	[b]274.7
			-423	329.6	(c)	(c)	6	(c)
				329.9	(c)	(c)	6	(c)
				[b]329.8	---	---	[b]6	---
	790° F for 20 hr	Hoop weld	70	(c) ↓	(c) ↓	(c) ↓	(c) ↓	256.8
								259.8
								[b]258.3
			-320	(c) ↓	(c) ↓	(c) ↓	(c) ↓	246.0
								258.2
								[b]252.1
			-423	(c) ↓	(c) ↓	(c) ↓	(c) ↓	189.9
								180.5
								[b]185.2

[b]Average value.
[c]No data taken.

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